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Hybrid Lithography in SU-8: A Masked-Based Photolithography and Direct Laser Writing Technique

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Hybrid Lithography in SU-8: A Mask-Based Photolithography and Direct Laser Writing Technique



A Senior Project

Submitted to the Division of Science, Mathematics, and Computing

At Bard College

By

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Annandale-on-Hudson, New York

May 2017

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Dedication

To my loving parents, Michael and Jennifer Clifton, I dedicate this project to you.

Acknowledgments

First and foremost I'd like to thank my project advisor, Dr. Chris LaFratta, without whom this project would not be possible.

Abstract

Presented here is a straightforward and cost-effective hybrid lithography technique. The process uses mask-based photolithography to pattern coarse features and direct laser writing (DLW) to customize features as small as 6 μm using a 20 \times 0.75 NA objective in SU-8, a widely used and commercially available photoresist. SU-8 doped with fluorescein (SU-8F) enabled its use for hybrid lithography because it shows contrast following exposure, as areas exposed absorb in the 450 nm region and unexposed areas do not. On average, DLW features written in SU-8 were 4.7 μm wider than features written in SU-8F. The DLW optical microscope was modified with a 450 nm filter to illuminate the sample, which caused mask-pattern features to appear opaque. The addition of fluorescein to SU-8 decreases its the ability to adhere to a silicon substrate. Using hybrid lithography, we were able to precisely pattern an interdigitated electrode in between two 110 μm leads on a silicon wafer.

1.0 Introduction

1.1 Mask-Based Photolithography

Mask-based photolithography is a method of lithography that utilizes a photoresist to transfer a pattern onto a given substrate. Photoresists can be characterized as either negative or positive tone. Upon exposure to the UV light, negative tone resists, which are often utilized in microfluidic and lab-on-a-chip fabrication, crosslink and polymerize, strengthening the polymer so that it is insoluble in the developing solvent. Positive photoresists are widely used in the microelectronics industry and undergo chain scission upon treatment with UV light, a process that cuts the side and main polymer chains, leaving exposed regions susceptible to the developing solvent.

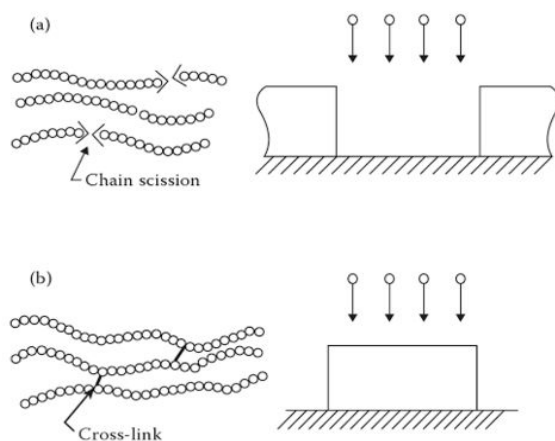


Fig. 1.1: (a) Polymer chain scission of a positive resist and (b) chain cross-linking of negative resist¹

The photoresist is purchased as a highly viscous liquid, and undergoes UV exposure after it is deposited onto a substrate, and then development (Figure 1.2). The substrate is dependent on the application. Microfabrication was conceived on pursuance of enhancing integrated circuit

(IC) manufacturing, encouraging the use of silicon wafer substrates within photolithography due to its semiconductive properties. In addition, silicon's ability to be chemically etched, a process which uses wet chemistry to selectively remove desired areas of the wafer, made it an ideal substrate during the origins of microfabrication and remains to this day.

Sixty years ago the first commercial IC was manufactured by Fairchild Semiconductor using a photomask to pattern a large series of transistors onto a silicon wafer. Technology within this time period only permitted the patterning of structures with resolution no better than 5 μm , but advancements in photolithography have led to the ability to obtain structures on the nanometer scale. Also recognized as UV lithography, mask lithography is the conventional method of the few pattern transfer fabrication techniques as it possesses the potential to create features in the millimeter to nanometer range. Moreover, the popularity of mask lithography is widely due to its ability to mass produce parallel patterns over a large area in the matter of seconds. This photochemical process is almost the exclusive means of manufacturing within the IC industry.

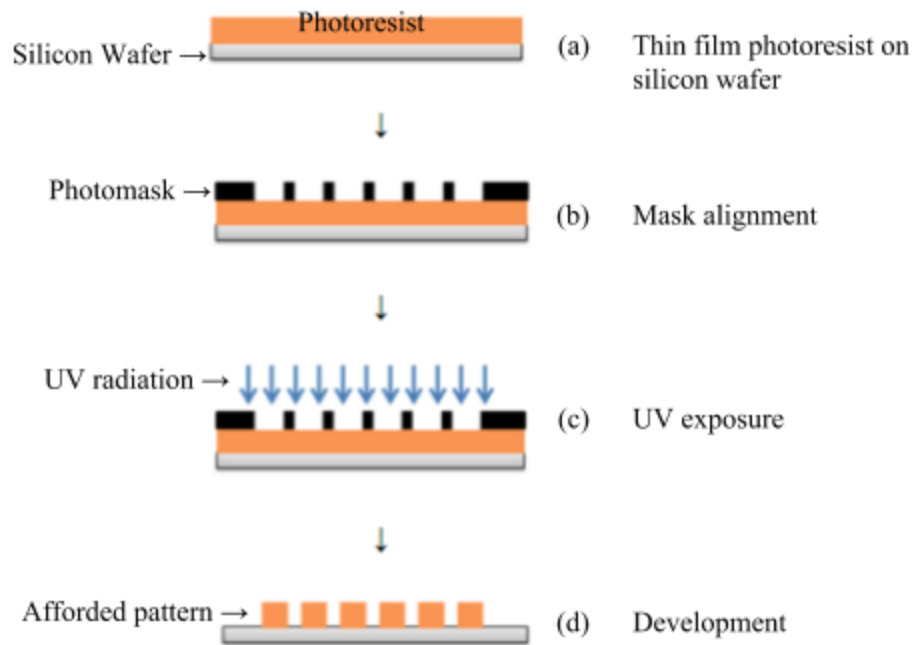


Fig. 1.2: Schematic of conventional mask-based photolithography using a negative-tone photoresist

The many attributes for which mask lithography is celebrated are ironically what lead to the drawbacks of the practice. While masks can pattern very high resolution structures, the cost of the photomasks increase as their corresponding feature sizes decrease, and frequently cost \$1,000's of dollars. Additionally, photomasks retain exact patterns, limiting researchers and manufacturers to the single pattern of the mask. Researchers within the past two decades have been advancing the field of nanofabrication due to the practicality of smaller scale technologies. The ability to resolve features within the most local, lateral dimensions creates smaller and faster computers, along with pushing for more environmentally and economically responsible chemistry by limiting both waste and resources employed.² Finally, UV lithography is a practical method for patterning two-dimensional features, but becomes less sensible for fabricating in

three dimensions. This defeats the use of conventional lithography within the field of biosensors, whose sensitivity and throughput are enhanced with respect to the increased surface area.²

1.2 Direct Laser Writing

As mentioned before, scientists have developed alternative methods of pattern transfer to combat the drawbacks of conventional mask lithography. Some of which include electron-beam lithography, one-photon polymerization direct laser writing (DLW), the primary method of microfabrication in the LaFratta Lab, and two-photon polymerization DLW. DLW utilizes an optical microscope to tightly focus a laser beam, subsequently patterning a photoresist through a photopolymerization reaction. One-photon absorption DLW uses a continuous wave laser to polymerize the photoresist, and can afford both coarse and extremely narrow linewidths depending on the user's desire. The invention of the pulsed femtosecond laser enables researchers to create three-dimensional, polymeric micro- and nanostructures using a motorized microscope stage moving in the x, y, and z. The novelty of this approach is the local excitation of the photoresist at only the focal point, simultaneously absorbing two photons of light. An example of the laser in practice during 2PP-DLW is a titanium-sapphire laser, whose pulses at femtosecond durations permit the phenomenon of two photon polymerization. The probability of a molecule of photoresist simultaneously absorbing two photons is proportional to the intensity of the laser beam squared (Figure 1.3).² This facet of DLW makes it an exceptional method of lithography as scientists in recent years have shown great interest and motivation in fabricating

micro- and nanostructures composed of more complicated geometrical systems.

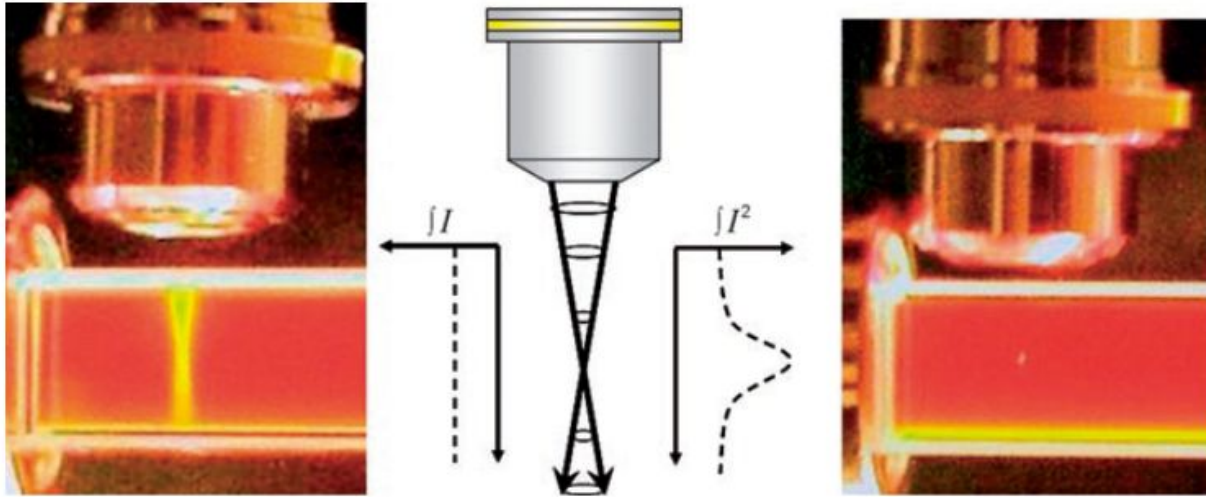


Fig. 1.3: The left image shows the continuous wave 1PP-DLW laser focused through a microscope objective lens into a solution of a fluorophore. The middle image shows the probability of a molecule absorbing one photon during 1PP-DLW is proportional to the intensity of the input beam, and the probability of absorbing two photons during 2PP-DLW is proportional to the intensity of the input beam squared. The right image shows a femtosecond pulsed 2PP-DLW system focused at only the focal point of the objective lens.

Both one-photon and two-photon DLW offer the ability to pattern microstructures like the ones produced through conventional lithography (Figure 1.4). DLW systems eclipse their conventional counterpart by enabling the user to create custom features that are unique compared to a photomask. Furthermore, 2PP-DLW and its ability to write in three dimensions allows the practice to be used within an immense range of applications that cannot be offered through conventional lithography. Despite the novelty of 2PP-DLW, the application has its drawbacks compared to 1PP-DLW, such as their substantial difference in cost. The price of a Ti:sapphire femtosecond laser can cost an upwards of \$120,000 (Thorlabs), compared to the \$185 spent on the 405 nm laser diode (Thorlabs) used by the LaFratta lab for microfabrication. Two-photon DLW is also less advantageous when desired patterns are larger features instead of on the

nanoscale. The localized pulse of the femtosecond laser makes patterning said features very time consuming. The primary concern within DLW is its prolonged process, making it impractical for mass production of ICs and lab-on-a-chip technologies. This project presents a cost-effective and time-saving approach to microfabrication in the SU-8, through the combination of conventional lithography to pattern coarse features and DLW for microscale features.

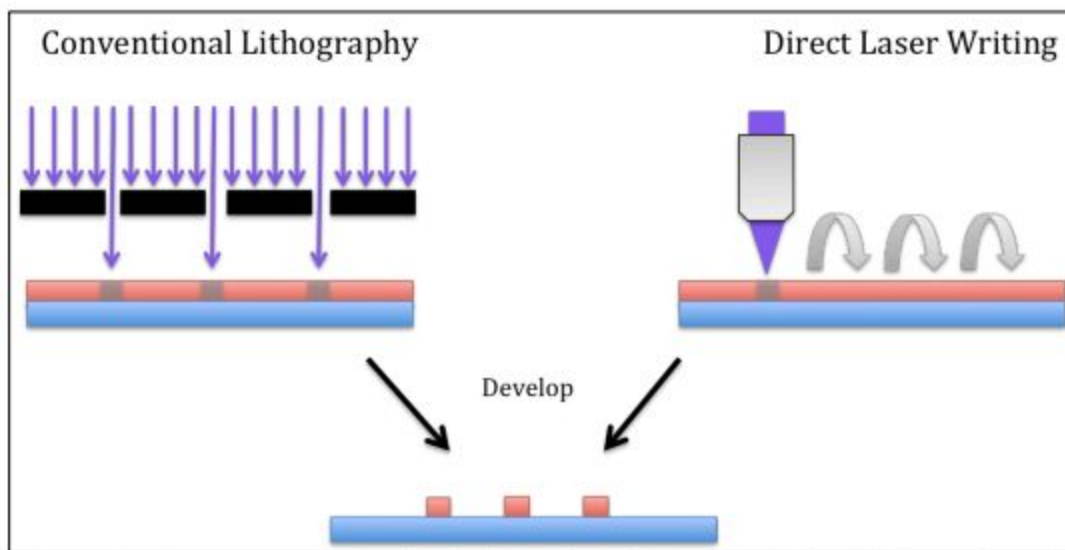


Fig. 1.4: Schematic comparing conventional lithography and DLW lithography and their ability to transfer patterns into a photoresist. The photoresist shown above is negative-tone.

1.3: *SU-8 2005*

SU-8 is a negative-tone, epoxy-type photoresist most sensitive to 365 nm radiation. It comes in a wide range of viscosities and subsequent film thickness after spin coating; SU-8 2000 producing the thinnest film, SU-8 2005 being slightly thicker and so on. SU-8 affords high aspect ratio structures due to its low optical absorption in the UV window. As an exceptionally chemically resistive material having low Young's modulus, the transparent and biocompatible

resist is an exemplary material for the micro- and nanofabrication of MEMS and bioMEMS, cantilevers, optical waveguides, microfluidic channels and more.³

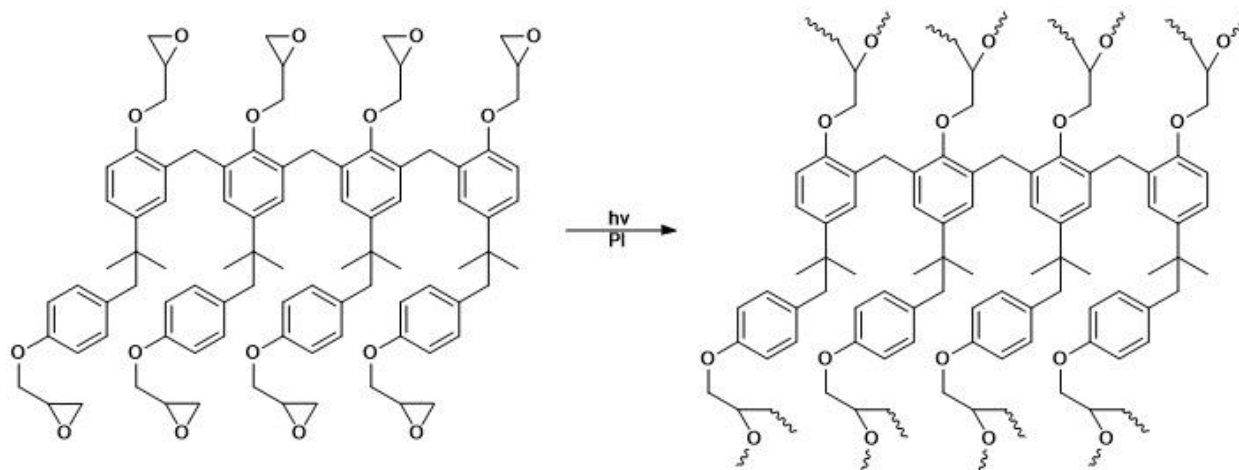


Fig. 1.5: Reaction scheme showing the photochemical reaction between SU-8 and 365 nm radiation. Upon exposure, the photoacid generator yields a Bronsted acid which initiates cationic polymerization of the epoxides.

SU-8 is an ideal photoresist for micro- and nanofabrication during conventional and DLW lithography processes. Unfortunately, it is least optimal when the two techniques are combined, as SU-8 is transparent prior to UV exposure, and remains so afterward. Moreover, this prohibits the ability to confidently align the sample on the DLW optical microscope after the conventional lithography step, making it improbable to accurately pattern the microscale features in correlation to the coarse features. How can one obtain visible contrast between exposed and unexposed regions of the photoresist in order to make hybrid lithography in SU-8 a viable method of microfabrication?

1.4: Hybrid Lithography

Outside of the LaFratta lab, researchers are studying the combination of conventional lithography and other microfabrication techniques. Kristensen et al. developed a process integrating electron beam lithography and UV lithography. In order to enable accurate alignment of their sample, Kristensen et al. created alignment marks on an SU-8 2000 covered silicon wafer layered in thermal oxide through reactive ion etching. E-beam lithography is then used to create structures on the micro- and nanoscale, followed by UV lithography to pattern larger features.⁴

Eschenbaum et al. integrates UV lithography to pattern in 2-D followed by two-photon DLW to pattern 3-D features. The group first spins rhodamine 6G doped SU-8 onto a glass coverslip which undergoes mask exposure and subsequent development in PGMEA. A film of pure SU-8 2050 is then spun onto the developed dye doped 2-D sample and is subjected to two-photon DLW 3-D patterning.⁵

The primary goal of this project was to determine a method of producing contrast between exposed and unexposed SU-8. This would enable the coarse features patterned through conventional lithography to be visible on the optical microscope for subsequent one-photon DLW microfabrication. It was originally hypothesized that doping SU-8 with a fluorescent dye may lead to this desired contrast, as photobleaching would occur in areas exposed to high energy UV radiation. The sample would then be illuminated through exposing it to radiation of the dye's excitation wavelength, causing the regions unexposed to UV radiation to fluoresce, and all that was originally exposed through the mask is unexcited.

The dye chosen was fluorescein, as it is readily bleached. The issue at hand is if fluorescein were to photobleach easily it would not be successful as a fluorescent dye. The

photodegradation of fluorescein doped SU-8 (SU-8F) was then tested to determine if the dye would be effective at producing contrast after exposure to UV radiation. It was seen that after UV exposure, the addition of fluorescein had transformed from a colorless film into a green-yellow film. Literature showed that fluorescein acts as an indicator, changing color in the presence of acid.⁶ This makes fluorescein an excellent candidate to promote visible contrast during the hybrid lithography process, as a photoacid is generated within SU-8 upon exposure to UV radiation. Furthermore, the green-yellow color of exposed SU-8F does not fade over time, as samples from almost one year ago retain their color.

Presented in this thesis is a fast, cost-effective method of hybrid lithography in SU-8, utilizing less expensive photomasks to pattern coarse features followed by one-photon DLW for the fabrication of features on the microscale.

2.0 *Experimental Methods*

2.1 *Synthesis of SU-8F*

To an 25 mL erlenmeyer flask, covered in aluminum foil, was added fluorescein (1 mg, 3 μ mol) and SU-8 2005 (10 mL). The mixture was then sonicated for 15 minutes and allowed to sit overnight at room temperature to ensure a homogenous mixture has been achieved. The SU-8F mixture was kept in a drawer to ensure minimal contact with room lighting.

2.2 *Direct Laser Writing Setup*

The laser used to perform one-photon DLW on a photoresist deposited silicon wafer was an OBIS 405 nm laser (Coherent, Inc.). The sample was placed upside down onto the computer controlled XY stage (Proscan III, Prior Scientific) of an inverted microscope (IX71, Olympus). The laser emits 405 nm plane polarized light which passes through a halfwave plate and polarizing beam splitter that are both used to manage the laser power. The laser beam is then focused through a lens and travels through the pinhole which filters most of the laser light. Upon exiting the pinhole, laser beam is collimated through a second lens. The beam reflects off of a series of mirrors until it is aligned with the back aperture of the microscope objective. Prior to reaching the objective lens, the laser beam passes through a 50/50 beam splitter, transmitting 50 % of the beam to be focused through the microscope objective and reflects 50 % of the laser light perpendicularly (Figure 2.1). This 50/50 beam splitter is not normally interrupting the path of laser but is necessary for DLW on a silicon wafer. The typical substrate used by the LaFratta lab is glass, but do to the poor adhesion of SU-8 onto glass, silicon is the ideal substrate for hybrid lithography. Because silicon is opaque to visible light, the sample cannot be illuminated through

the microscope lamp. An outside, white light source (Cole-Parmer, 41723-Series High Intensity Illuminator) passes through a 450 nm bandpass filter (Thorlabs) illuminating the sample.

Exposed SU-8F has a high absorption in the 440 nm to 450 nm range, whereas unexposed SU-8F has negligible absorption within this spectral region. Therefore the coarse features patterned during conventional lithography appear black, and the rest of the sample appear an aqua blue color. The 450 nm light originally travels perpendicular to the path of the laser until reaching the 50/50 beam splitter which reflects half of the light into the microscope object. The 450 nm light then reflects off of the silicon wafer and back down into the detector.

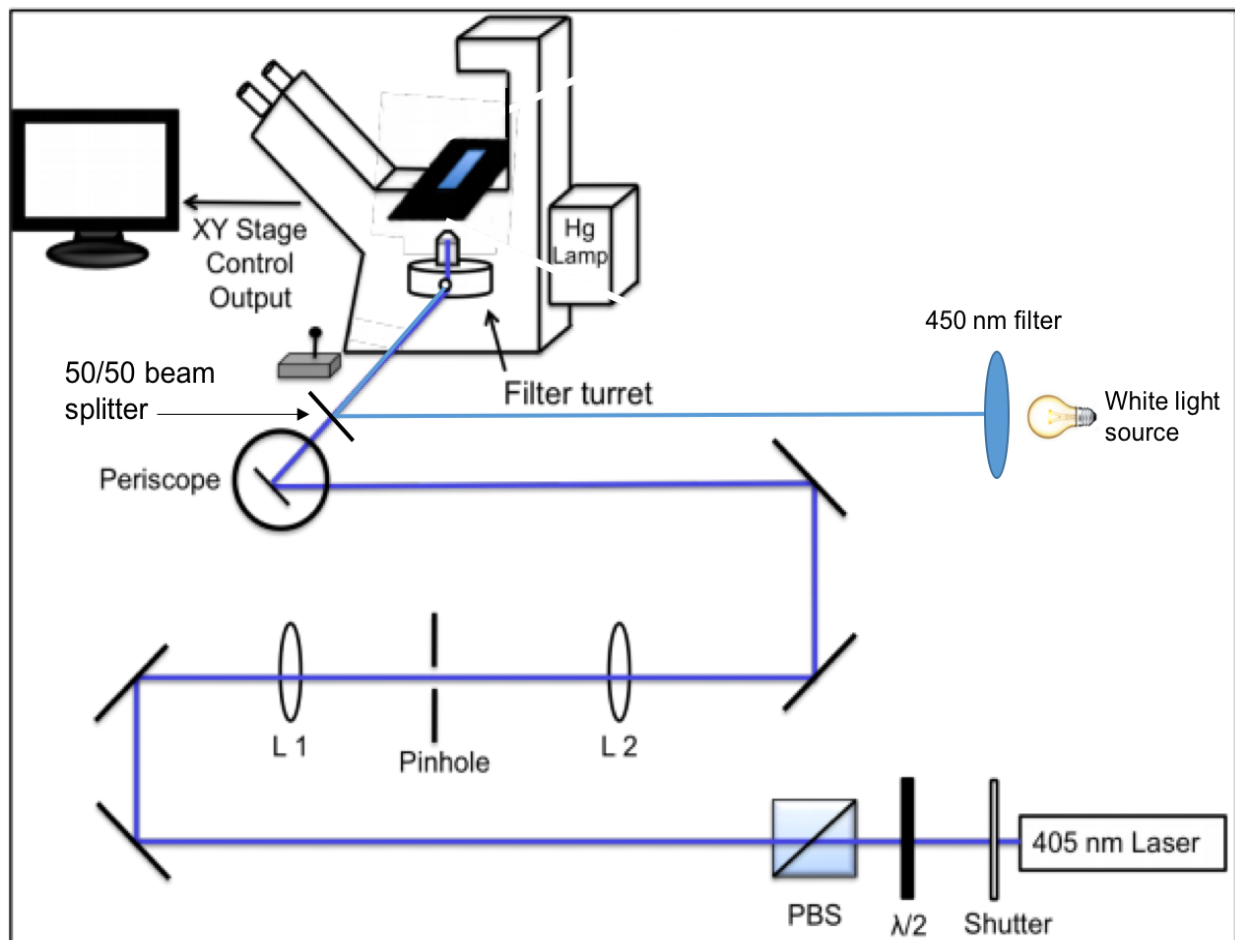


Fig. 2.1: Schematic of the laser path for direct laser writing on a silicon substrate. 450 nm light is directed into the sample by a 50/50 beam splitter illuminating the sample, affording visible contrast between unexposed and exposed SU-8F.

2.3 *Sample Preparation and Hybrid Lithography Procedure*

A silicon wafer was subjected to O₂ plasma cleaning for 1 minute. Any residual dust on the silicon wafer was removed by argon gas. The wafer was then placed onto the chuck of the spin coater (Model WS-400B2-6NPP/LITE, Rev. MS). Pipetted onto the center of the wafer was ~1 mL of SU-8F to be spin coated. The resist was made to cover the entire wafer by spinning it at 800 rpm for 30 seconds. To ensure a thin film of 5 μ m - 10 μ m thickness, SU-8F was deposited onto the wafer and spun at 1500 rpm for 105 seconds. The final stage of spin coating was a slow spin at 300 rpm for 15 seconds. This was done as a safeguard to prevent damaging the flat, smooth film achieved during the rapid spin, as an abrupt halt during this step may give a nonuniform film. The SU-8F covered silicon wafer was placed into a petri dish and covered in aluminum foil. SU-8 2005 (Microchem) and contains ~1 % of the developing solvent, PGMEA, due to its photosensitivity. To remove the solvent the wafer was prebaked at 65°C for 3 minutes and 95°C for 9 minutes. The temperatures were recorded using FlirONE for iOS. The mask was aligned onto the silicon wafer using the eye. The lined features of the mask were placed perpendicular to the straight edge of the wafer, on top of which was clamped a block of quartz to ensure the space between SU-8F film and mask was minimized for the homemade UV lithography apparatus. SU-8F was exposed for 60 s to 365 nm radiation by placing the entire apparatus under a Hg lamp for coarse feature patterning. The sample was then placed into the aluminum foil covered petri dish and brought to the laser for subsequent microfabrication using one-photon DLW. SU-8F was patterned using laser powers of 500 μ W - 1500 μ W, recorded after

the pinhole, in intervals of 100 μW . Using the 20 \times 0.75 NA objective (Olympus, UPlanSApo), lines were drawn at 5 $\mu\text{m}/\text{s}$ - 50 $\mu\text{m}/\text{s}$ in intervals of 5 $\mu\text{m}/\text{s}$ at each laser power. After both UV and 405 nm laser exposure, the sample was post-exposure baked at 65°C for 1 minute and 95°C for 12 minutes. During this step the cleaved epoxide rings crosslink and adhere to the silicon substrate. The hybrid lithography sample was allowed to cool at room temperature for 2 minutes. Using tweezers, the sample was placed in the developing solvent propylene glycol methyl ether acetate (PGMEA) for 20 seconds. The sample was removed and this process was continued until the sample had been subjected to the developing solvent for a total of 2 minutes. Afterwhich, the sample was rinsed using isopropyl alcohol. If a cloudy, white film formed upon rinsing in IPA, it meant the sample was underdeveloped, and needed to undergo further development until no film formed over the wafer. Fully developed samples that were SEM worthy were sputter coated in gold (Cressington, Model 108 Sputter Coater) and imaged through SEM (Tescan, MIRA 3). All other sample were imaged and linewidths measured through DLW optical microscope and ProScan GUI.

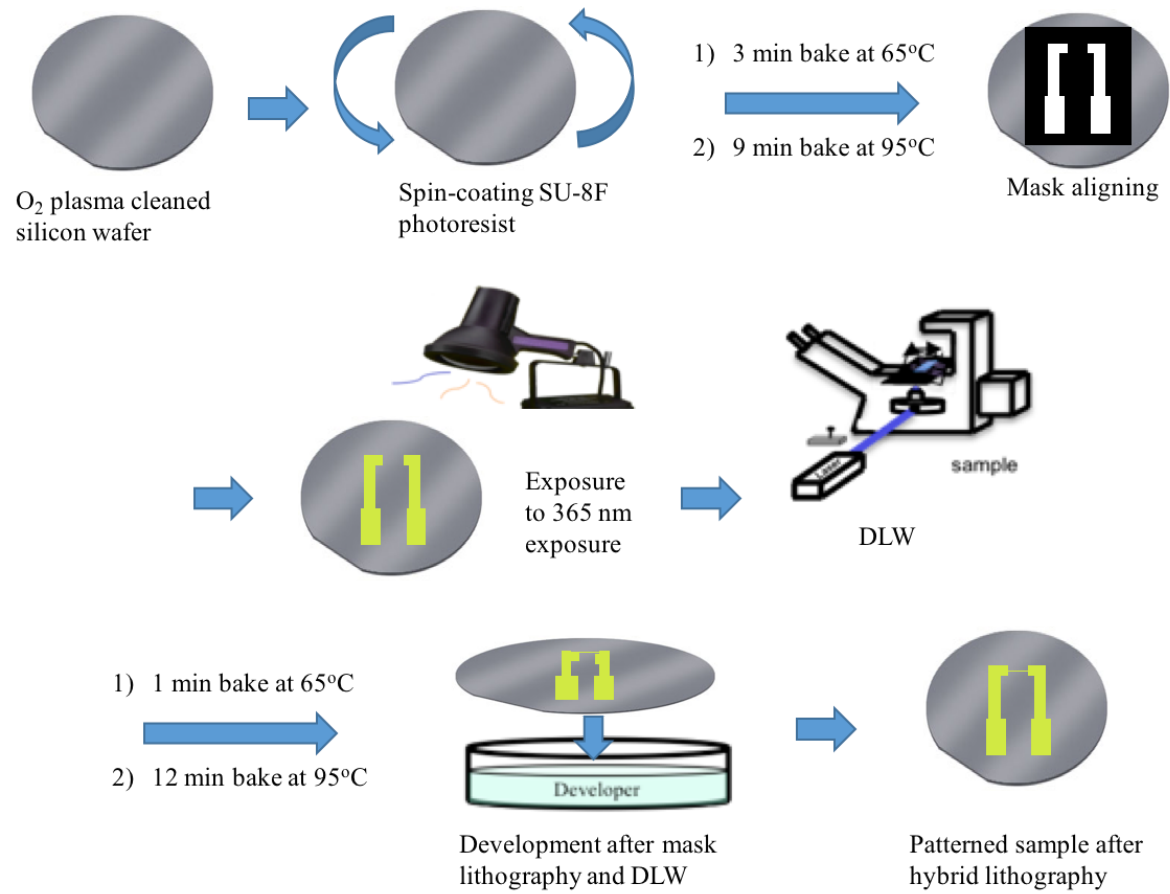


Fig. 2.2: Schematic of hybrid lithography procedure combining conventional mask lithography and one-photon DLW.

3.0 Results and Discussion

The addition of fluorescein to SU-8 2005 allowed for features patterned through conventional lithography to be visible to the eye as unexposed SU-8F film is colorless and the exposed SU-8F film is green-yellow. UV-vis spectra was taken of SU-8F both before and after exposure to 365 nm radiation. The spectra showed that exposed SU-8F absorbs in the 450 nm range, unlike unexposed SU-8F who has negligible absorption in this region. Furthermore, illuminating the SU-8F sample with white light passed through a 450 nm filter allowed the features patterned through mask lithography to be clearly visible on the DLW microscope. Microstructures patterned using through DLW were also visible on the optical microscope in real time.

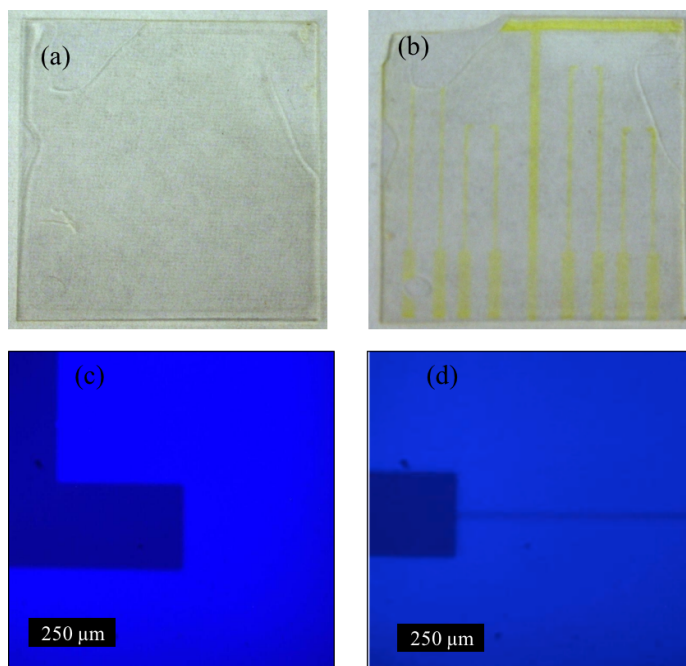


Fig 3.1: (a) Optical image of unexposed SU-8F on a 25 x 25 mm coverslip. (b) Optical image of exposed SU-8F shows contrast between unexposed (colorless) and mask-exposed (green-yellow) SU-8F. (c) Micrograph of mask-exposed SU-8F showing contrast between unexposed (blue) and

mask-exposed (opaque, dark blue) SU-8F taken with 4 × objective. (d) Micrograph of line drawn through 4 × objective.

When comparing SU-8 and SU-8F, the addition of fluorescein had negligible effect on the photoresist and its ability to adhere to the silicon substrate during the conventional lithography step, nor did it affect the development as both resists needed 120 seconds to 140 seconds in PGMEA.

However, the addition of fluorescein negatively affected SU-8's ability to adhere to the silicon wafer during the DLW step, as lines drawn with the 405 nm laser required greater powers and lower speeds to promote adhesion. It was thought that incorporating fluorescein into SU-8 elevated the boiling point of the developing solvent resulting in the poor adhesion of the doped resist, but increasing the prebake times showed little to no affect on the adhesion of SU-8F. Additionally, increasing the post-exposure baking times, especially at 95°C, was attempted to promote polymer cross-linking and consequently strengthening adhesion. Unfortunately, adhesion was not enhanced, and when the sample was postbaked for longer than 15 minutes at 95°C, the sample appeared cooked, having a ring around each of the fabricated structures.

The hybrid lithography technique faced further complications during the DLW step in part due to the 50/50 beam splitter implemented to direct the 450 nm light used to illuminate the sample. Theoretically, half of the laser beam intensity is transmitted through the beam splitter, but human error leads to less than 50 % of the laser beam intensity is directed into the sample, requiring much higher laser powers to yield adhesion. This drawback could be accounted for if both mask alignment and sample alignment on the microscope stage were done through an automated process. If so, the 450 nm illuminating light could be sent directly into the back aperture of the microscope objective in order to locate an origin for DLW. The program could

then be set to fabricate a desired pattern, allowing the entire process from this point on to be done in the dark.

An additional drawback was that the sample could never be completely flat on the microscope stage, presumably due to a nonuniform film. Consequently, as the motorized stage moved the laser beam would go out of focus, requiring a hand on the fine adjustment of the microscope to ensure the beam was focused.

Despite imperfections within the DLW step, features with linewidths as small 6 microns were fabricated in SU-8F. Lines fabricated through DLW achieved best adhesion at high powers, and lost adhesion ability as powers decreased. Furthermore, microstructures adhered most to the silicon wafer at slower writing speeds, but lost adhesion as the speeds were increased. Identical DLW experiments attempted in pure SU-8 afforded adhesion to the silicon substrate at each parameter, but produced thicker linewidth structures.

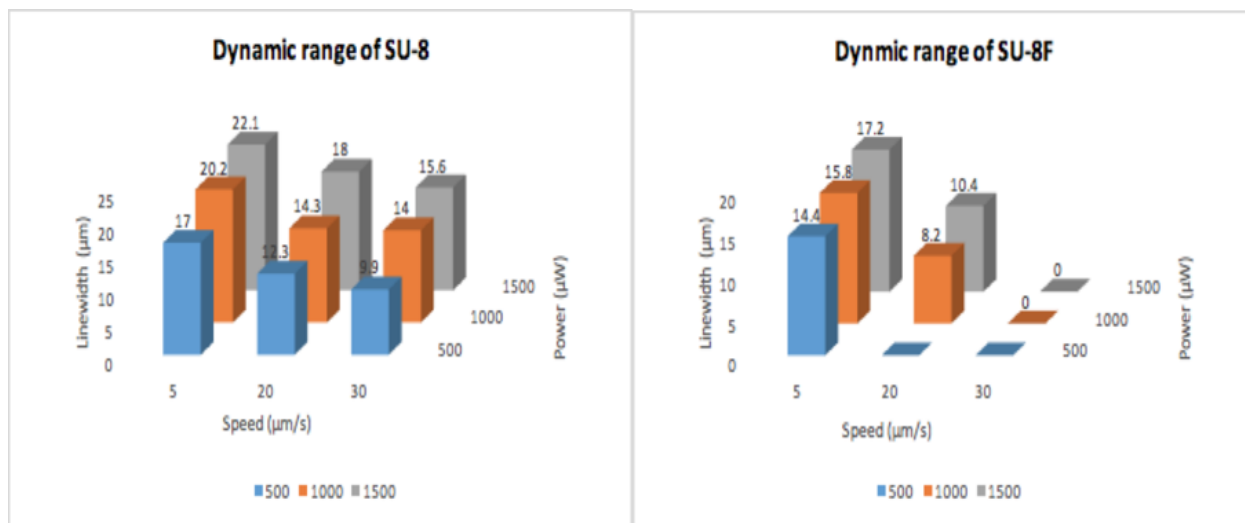


Fig. 3.2: The data above displays the average linewidth as a function of power and speed of the 405 nm using a 20×0.75 NA lens. Linewidths of SU-8 were on average 4.7 μm thicker than those drawn in SU-8F.

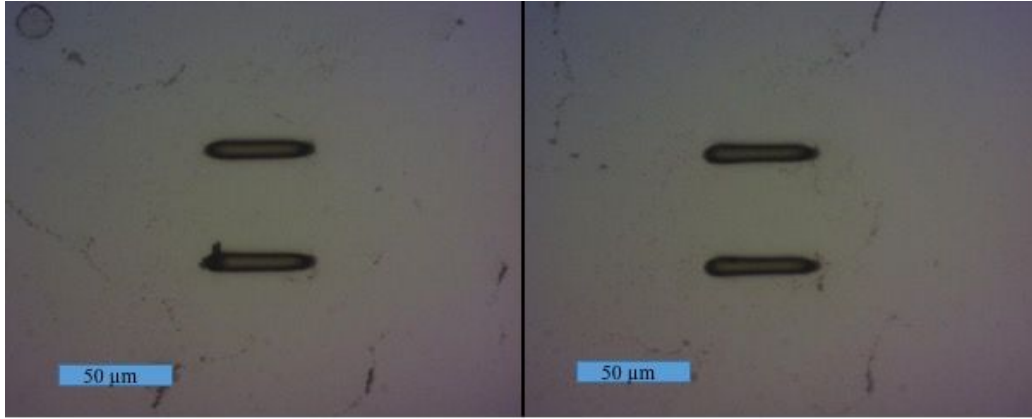


Figure 3.3: The left micrograph shows DLW lines with 8.5 μm linewidths, drawn at 500 μW and 10 $\mu\text{m}/\text{s}$ through the 20 \times 0.75 NA objective lens. The right micrograph shows DLW lines with 6.0 μm linewidths, drawn at 1000 μW and 25 $\mu\text{m}/\text{s}$ through the 20 \times 0.75 NA objective lens.

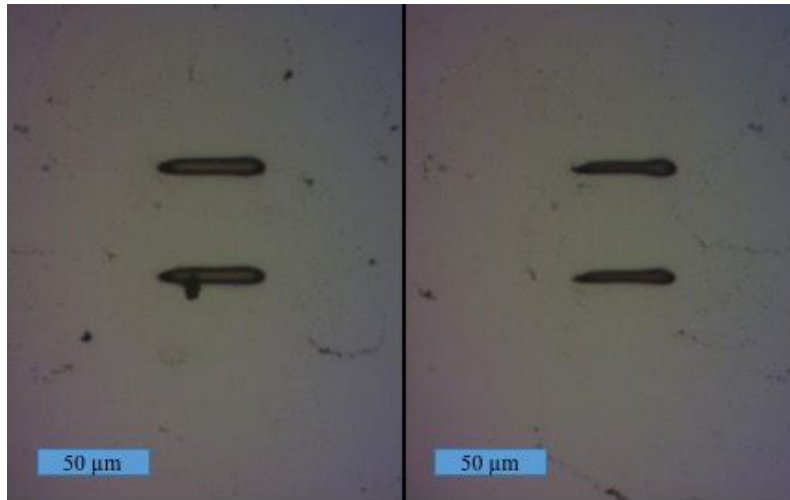


Figure 3.4: The left micrograph shows DLW lines with 7.5 μm linewidths, drawn at 500 μW and 25 $\mu\text{m}/\text{s}$ through the 20 \times 0.75 NA objective lens. The right micrograph shows DLW lines with 6.0 μm linewidths, drawn at 1250 μW and 30 $\mu\text{m}/\text{s}$ through the 20 \times 0.75 NA objective lens.

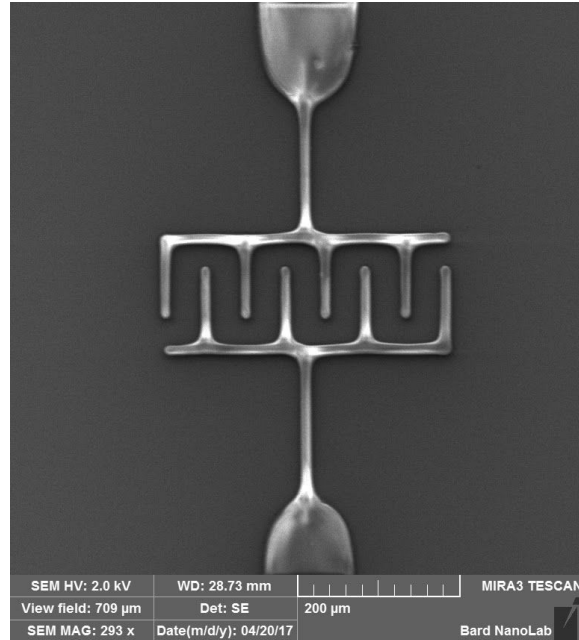


Figure 3.5: An SEM image of interdigitated electrode fabricated between two 110 μm width leads through DLW.

4.0 Conclusion

In conclusion, we have developed a cost-effective, time-favorable method of hybrid lithography in the popular negative-tone photoresist, SU-8, through combining the likes of masked-based photolithography and one-photon direct laser writing. Fluorescein doped SU-8 proved successful for this project as masked-exposed areas of the photoresist were made visible on the DLW optical microscope through illuminating the sample with 450 nm light. This hybrid lithography process is optimal for patterning coarse features through conventional lithography followed by DLW to fabricate structures as small as 6 μm on the wafer scale, can be made more practical if computer automated processes were involved during the mask alignment and microscope stage alignment steps.

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